DEVELOPMENT OF A PROTOTYPE IFE FOAM CAPSULE TARGET

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We Were Asked To Produce A Hollow Plastic Foam Shell With An Overcoat.

- Design is specified by NRL.
- 4 mm diameter shell
- 300 micron polymer foam wall
  - CH polymer
  - ~ 1 micron cell size
  - 10 - 100 mg/cm³
- 1 micron polymer overcoat
- 0.03 micron metallic coating
Similar Overcoated Foam Targets Had Been Made.

- ILE produced 0.8 mm diameter, 30 micron wall shells from an acrylate foam system. (Takagi, et al)
- LLNL/Schafer produced 2 mm, 100 micron wall shells from both the acrylate and resorcinol-formaldehyde foam systems.
- There are two very significant differences in the NRL IFE design.
  - The foam polymer can contain only carbon and hydrogen, no oxygen.
  - The diameter of the shell is doubled.
First We Created A Suitable CH Foam.

- Divinyl benzene ($\text{C}_{10}\text{H}_{10}$) can be used to make foams within a density range of 10 - 250 mg/cm$^3$.
- The process is analogous to the acrylate foams.
- At 10 mg/cm$^3$ the average cell size appears to be about 1.6 microns.
- The foam is cast to near net shape, shrinkage is very minimal.

This SEM shows the cell size and morphology of a 10 mg/cm$^3$ foam.

This photograph shows a 14 mg/cm$^3$ foam in isopropanol. It was cast with tabs at each end.
The DVB Foam Has A Unique Set Of Properties.

Density Range of Low Density Foams

- Si Aerogel
- Hypercrosslinked
- Acrylate (TMPT)
- Resorcinol-Formaldehyde
- HIPE Polystyrene
- TPX
- DVB

Cell Size Range

- Si Aerogel
- Hypercrosslinked
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- TPX
- DVB

= CH foam
= IFE region
To Make Shells, We Microencapsulate Using A Droplet Generator.

- **Inner Water Phase:**
  - D$_2$O / H$_2$O Blend
  - flows through innermost needle.

- **Organic Phase:**
  - dibutyl phthalate, DVB, radical initiator
  - flows through second needle.

- **Stripping Phase:**
  - 5 % PVA in H$_2$O
  - strips off organic/water drop.
Our Droplet Generator Is Made From “Off the Shelf” Parts.

- System consists of two syringe pumps, stripping fluid pump, triple orifice, and rotary evaporator.
- System is readily adaptable to making shells with different diameter and wall thickness by changing flow rate and needles.
The DVB Must Polymerize Quickly Or The Complex Drop Will Be Destroyed By Rotation.

- Drop must gel 15 minutes after formation to avoid breakage.
- Temperature, initiators, and addition of other CH monomers were investigated to hopefully hasten gelation.
- Monomer concentration is most importantly factor.
- To compensate for the slow gelation, we must start the polymerization before microencapsulation.
The Keys to Making a Spherical Uniform Shells Are Density, Interfacial Tension, and Agitation

- Much Work has focused on density matching.
  - Density matching helps center the inner and outer drop, but density is a function of temperature.
- Influences of interfacial tension and agitation need to be studied further as will be seen.
Initial Density Matching Results Were Very Promising.

- We measure the densities of the oil phase and internal water phase at ambient temperature.
- The polymerization occurs at 85º C, the density of the organic phase will be reduced more than the density of the water phase.
- Characterizations were done here at General Atomics.
Additional Density Matching Results Reminded Us How Difficult This May Be.

- After more data was collected in the 0.010 to 0.012 g/cc range, the trend appeared to be less clear. Optimum now appears to be about 0.019 g/cc.
- If the optimum was exceeded a “V” shape would be expected when the data is plotted in this manner.
Reinterpreted Density Matching Data Gives Two Possible Answers.

- We need to examine the results for a greater than .015 density difference to clarify the situation.
- Other factors may have dominated the results - temperature, interfacial tension, agitation.
Temperature Control Is Being Improved.

- Temperature in gelation flask was found to vary from 65-73 °C.
- A temperature controller was obtained to minimize temperature fluctuations.
- DVB polymerization experiments using UV initiation performed.
- Polymerization achieved, but too slow to be useful.
To Overcoat The Polymer Shells, We Are Using A Technique Developed At ILE.

• ILE overcoated acrylate shells, we previously overcoated RF shells using an interfacial polymerization.

• The reaction occurs at the interface between the water and organic phases.

• It is a multi-step process that requires time and labor.

• There are a variety of reactants that can be used to optimize the resultant wall.
For Example, The Wall Thickness Is A Function Of The Polymer Crosslinking.

- Typical reactants are polyvinyl phenol (PVP) and acid chlorides.
The Surface Finish Of The Overcoat Is Critical.

- The intrinsic surface finish produced by the PVP and triamine with isophthaloyl dichloride looks very good.
We Can Confirm Overcoating on “Wet Capsules” Using Confocal Microscopy.

- Confocal image of shell
- 900X magnification
- Image is 100 microns across
- All of these features are submicron in depth.

Uncoated Shell

Coated Shell
We Need To Overcome Shell Characterization Issues.

• Shells do not ship well - ~25% broken or cracked after shipment.

• ~95% Broken or cracked after exchanging to DBP for characterization (estimates and image from GA). Handling needs to be minimized.

• To overcome these two problems we will begin characterizing the capsules at Sandia.
We Will Combine Optical Characterization With Radiographic Characterization.

- This is a radiograph of a DVB foam shell.
- System is being modified for foam needs: optimizing energy and adding rotation.
- System currently has ~28 micron resolution.
- Also designing system with ~10 micron resolution – Planned for FY04.
There Is Much Work That Needs To Be Done.

- Determine optimal density matching and its significance.
- Determine advantage of using PVA or PAA system.
- Determine methods to reduce shell cracking.
- Develop x-ray radiography characterization system.
- Develop wet characterization system at Sandia.
Nevertheless, We Have A Prototype IFE Capsule.

- DVB is an appropriate polymer for the foam (density, cell size, elemental composition).
- It has been microencapsulated to produce 4 mm diameter capsules with densities between 50-100 mg/cm³.
- It is chemically compatible with a polymer overcoating process that can produce walls of the appropriate thickness and surface finish.